PART 1

INTRODUCTION

Chapter 6. Congener Pattern Matching of Data Collected for the Lake Michigan Mass Balance Project (LMMBP)

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1.6.1 Introduction

As part of the quality assurance (QA) process for the Lake Michigan Mass Balance Project (LMMBP), a LMMBP PCB Modeling Peer Review Conference was held on July 27-28, 2004 at the Crowne Plaza Hotel in Romulus, Michigan to review the LMMBP polychlorinated biphenyl (PCB) models developed for this study. One of the recommendations to come out of this peer review was to "Investigate congener patterns in air, water, fish, and sediment. How do these compare?" (Part 7, Appendix 1). In response to this question, the peer review response document states that: "The PCB patterns of multiple media will be compared to determine similarities and differences within and among media. This technique is commonly referred to as PCB fingerprinting or PCB signature recognition and has had mixed success in the past. This recommendation has minor implications to the modeling; however, it is a data analysis tool and has merit for data presentation and interpretation purposes. The relative percent of total PCBs represented by each congener will be computed and then expressed as a cumulative frequency plot for comparative purposes. These will represent data for an entire study period, will be tested with both mean and median values, and will be a composite expression of seasonal and spatial data. In addition, selected evaluation of pattern recognition using the LMMBP data set can be found in Kuehl (2002) and McCarty *et al*. (2004). Fingerprints will be calculated for sediment, water column (dissolved and particulate), vapor phase, wet and dry atmospheric deposition, and age 5-6 year-old lake trout signatures from the Saugatuck biota site. Atmospheric signatures will be based on a subset of all congeners because vapor phase data were computed by Keri Hornbuckle for the study, and over-lake concentrations were only calculated for the congeners that are being modeled at Grosse Ile. In addition, PCB patterns associated with water discharging from the Kalamazoo River near the Saugatuck biota site and other selected tributaries will be compared/contrasted to the lake water".

One of the main objectives of this analysis was to see if there was a correlation between congener patterns in the LMMBP biota boxes and the atmospheric, tributary, and sediment sources of congener contamination. To accomplish this, congener pattern matching analysis was expanded from that originally suggested in the peer review modelers' response. This included congener pattern matching for all 11 major tributaries, as well as a comparison of atmospheric inputs for all 10 surface segments defined in the LM2-Toxic model. In addition, ages 5-6 year-old lake trout were evaluated in Sturgeon Bay, Sheboygan Reef, and Saugatuck biota boxes (Figure 1.6.1), instead of just in the Saugatuck biota box. Multiple year classes (age 2, age 3, and age 9), of lake trout were analyzed for the Saugatuck biota box, to see if there were differences in the congener patterns of different aged lake trout.

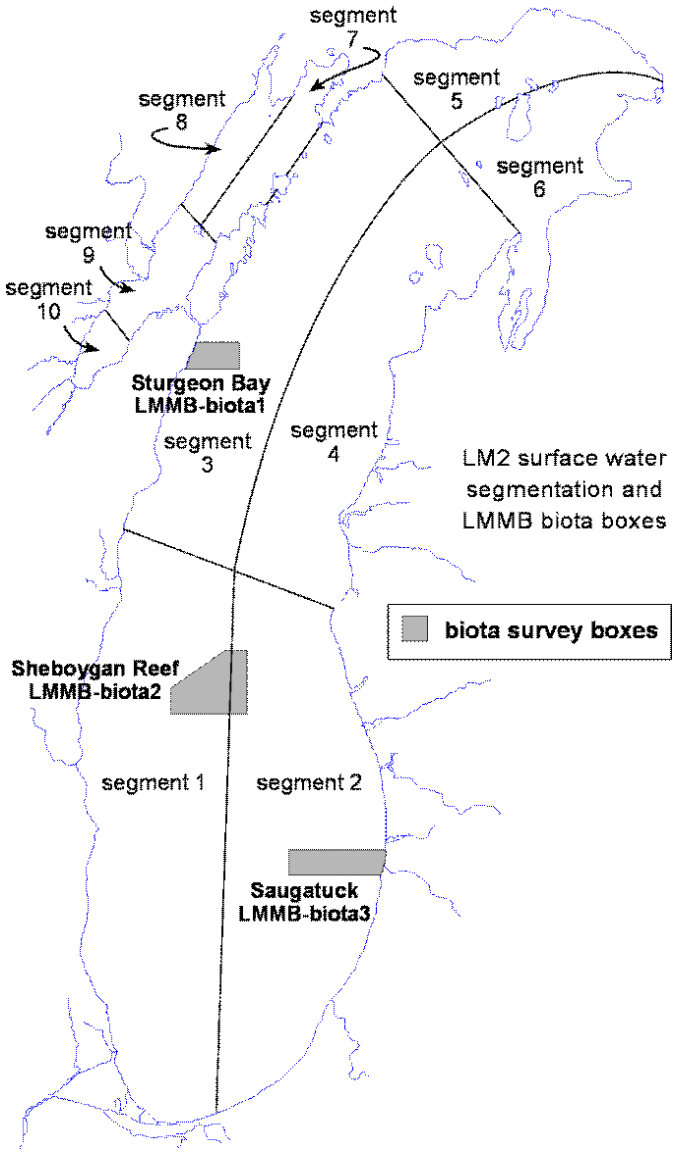

Figure 1.6.1. LM2 surface water segmentation and LMMBP biota boxes.

1.6.2 Analytical Approach

This exercise was not meant to be a rigorous statistical analysis of the data, rather it is strictly an empirical look at congener patterns in different media collected for the LMMBP. All available congener data were included in the analysis. Analysis was done for most of the media collected for PCBs during the LMMBP (atmospheric, tributary, lake water, sediment, and biota). Kuehl (2002) previously had done a comparison of congener patterns of the biota media (phytoplankton, zooplankton, *Mysis, Diporeia*, bloater chub, slimy and deepwater sculpin, alewife, rainbow smelt, coho salmon, and lake trout) collected for the LMMBP. Therefore, with the exception of lake trout, these biota media were not examined in the present analysis. For all of the analyses done for the present study, comparisons were made by plotting the congener patterns and doing a visual comparison for obvious similarities or differences.

1.6.3 Methodology

PCB congener analysis for the LMMBP were performed by a number of different principal investigators, using different instrumentation and techniques, which are detailed in the LMMB Methods Compendium (U.S. Environmental Protection Agency, 1997). These variation in methods may have had some impact of the comparison of samples from different media due to co-elution and congener detection differences from media-to-media.

All analyzed congeners were used to maximize the amount of data available. All analyses were done using Microsoft Excel spreadsheets. Congeners were ordered in each media by congener number, with co-eluting congeners ordered by the lowest co-eluting congener number (see Table 1.6.1) Data were analyzed by media, where the mean and median values were calculated for each congener. Cumulative frequency analysis was then performed on the means and medians for each congener. The percent frequency of each congener was calculated by dividing the means and median of each congener by the total sum of all the congeners in that media to give a percent value to each congener. These percentages were added together to give a cumulative frequency distribution which totals to one.

These data were graphed, and visual comparisons were done.

1.6.4 Results

1.6.4.1 Comparison of Modeled Congener Patterns to All Analyzed Congener Patterns

In an effort to make use of as many congeners as possible in this analysis, all congeners analyzed in each media were used rather than just the modeled

Table 1.6.1 Comparison of Congeners Available for Analysis in All LMMBP Media

Modeled Congeners	Available Congeners in All Media						
	Vapor Phase	Wet Deposition	Dry Deposition	Open Lake Water	Surficial Sediment	Tributary	Lake Trout
8+5	8+5	8+5	8+5	4+10	8+5	003	022
15+17	15+17	012	012	8+5	006	4+10	24+27
16+32	16+32	013	013	006	7+9	8+5	31+28
018	018	15+17	15+17	7+9	012	006	029
026	026	016	016	012	013	7+9	033
28+31	28+31	018	108	013	15+17	15+17	040
033	033	026	1026	014	016	16+32	41+71
37+42	37+42	31+28	31+28	15+17	018	017	042
044	044	032	032	016	019	018	044
049	049	033	033	018	021	019	47+48
052	052	037	037	019	022	022	049
56+60	56+60	042	042	021	24+27	24+27	052
066	066	044	044	022	025	025	56+60
70+76	70+76	049	049	024	026	026	063
074	074	052	052	025	31+28	31+28	064
77+110	77+110	56+60	56+60	026	029	033	066
081	081	066	066	027	032	37+42	70+76
92+84	92+84	70+76	70+76	31+28	033	040	074
085	085	074	074	029	37+42	41+71+64	77
087	087	077	077	032	040	044	81+87
089	089	081	081	033	41+71	045	082
099	099	92+84	92+84	37+42	042	046	083
101	101	085	085	040	043	47+48	92+84+89
132+153+105	132+153+105	087	087	41+71	044	049	085
118	118	089	089	043	045	051	091
123+149	123+149	099	099	044	046	052	095
163+138	163+138	101	101	045	47+48	053	097
146	146	132+153+105	132+153+105	046	049	56+60	099
151	151	110	110	47+48	051	063	101
170+190	170+190	118	118	049	052	066	105
172+197	172+197	123+149	123+149	051	053	66+95	107
180	180	163+138	163+138	052	56+60	70+76	110
187+182	187+182	170+190	146	053	063	074	114
195+208	195+208	172	151	56+60	064	77+110	118
196+203	196+203	180	170+190	063	066	082	119
201	201	187+182	172	064	70+76	083	123
		208+195	180	066	074	92+84	126
		196	187+182	70+76	77	085	128
		197	108+195	074	081	087	129
		201	196	77+110	082	089	131
		203	197	081	083	091	132+153
			201	082	92+84	095	134
			203	083	085	097	135+144
				92+84	087	099	137+176
				085	089	101	163+138
				087	091	105+132+153	141
				089	095	118	146
				091	097	123+149	149
				095	099	128	151
				097	100	132+153	156
				099	101	135+144	157
				100	105+132+153	136	158
				101	107	137+176	167

Table 1.6.1. Comparison of Congeners Available for Analysis in All LMMBP Media (Continued)

	Available Congeners in All Media						
Modeled Congeners	**Vapor Phase**	**Wet Deposition**	**Dry Deposition**	**Open Lake Water**	**Surficial Sediment**	**Tributary**	**Lake Trout**
				103	110	163+138	170+190
				105+132+153	114+131	141	171
				107	118	146	172
				114+131	119	149	173
				118	123+149	151	174
				123+149	128	158	175
				147+124	129	167	177
				128	130	170+190	178
				129	134	202+171	180
				130	135+144	172	187+182
				134	136	172+197	183
				135+144	137+176	174	185
				136	163+138	177	189
				137+176	141	178	191
				163+138	146	180	193
				141	151	187+182	194
				146	156	183	195
				151	157+200	185	203+196
				156	158	193	197
				157+200	167	194	198
				158	170+190	208+195	199
				167	202+171	203+196	200
				170+190	172	198	201
				202+171	173	199	202
				172+197	174	201	205
				173	175	206	206
				174	177	207	207
				175	178		208
				177	180		209
				178	187+182		
				180	183		
				187+182	185		
				183	189		
				185	191		
				189	193		
				191	194		
				193	208+195		
				194	203+196		
				208+195	197		
				196	198		
				198	199		
				199	201		
				201	205		
				203	206		
				205	207		
				206	209		
Total # Congeners:				207			
				209			
54	54	54	56	127	123	107	98

congeners. Because most of the comparisons were in the same media, this would give the maximum number of data points. However, there were some concerns when comparing data from different media. To see if this approach was feasible, an initial comparison was done to see if the congener pattern trends seen using only modeled congeners were different from the congener patterns seen when "all available congeners" were analyzed (Table 1.6.1). This analysis was done for a number of different media (vapor phase, wet and dry deposition, dissolved and particulate PCBs in lake water, surficial sediments, and 5-6 year-old lake trout).

The definition of "all congeners" varied somewhat, depending on the medium being examined. For dissolved and particulate water, sediment, tributary, and lake trout data, all congeners reported were used in this analysis. This number varied from a low of 98 congeners in the lake trout analysis to a high of 127 congeners in the open lake water samples. For the atmospheric samples, Keri Hornbuckle calculated over-water concentrations of only modeled congeners in vapor phase, as well as wet and dry deposition data (Table 1.6.1); therefore, the only congeners available in this media are the 54 modeled congeners. While over 100 congeners were available from Hornbuckle in the atmospheric wet and dry deposition data sets, only a subset were used because data needed to be aggregated by LM2-Toxic modeling segment. These aggregated data sets contained only 54 congeners of wet deposition data and 56 congeners of dry deposition data (Table 1.6.1).

This comparison was limited to the samples collected in or near the Saugatuck Biota Box, LMMBP-Biota3, for lake trout and surficial sediment, or to LMMBP modeling segment 2 (Figure 1.6.1), which contains the Saugatuck biota box, for the atmospheric and lake water samples. In addition, dissolved and particulate water data for the Kalamazoo River were included, because this is the tributary which discharges water closest to the Saugatuck biota box.

Cumulative frequency plots comparing all available congener data and modeled congener data were then created. This comparison showed that the trends in the different media were similar, whether modeled congeners or all available congeners were used (Figures 1.6.2-1.6.10). This is not too surprising because the 54 modeled congeners made up the bulk of the congener mass in all media sampled, accounting for an average of 74% of the mass across all media (77.3% of vapor phase PCB mass, 63.4% of wet deposition PCB mass, 73% of dry deposition PCB mass, 80.2% of tributary water PCB mass, 67.5% of dissolved lake water PCB mass, 77.2% of particulate lake water PCB mass, 84.7% of surficial sediment PCB mass, and 67.1% of lake trout PCB mass). Because these trends were similar, it was decided that all available congeners would be used in the data analysis, so that we could take advantage of the maximum amount of data points.

1.6.4.2 Comparison of Median to Mean Data

A comparison between mean and median values in different media was done to see how much results varied between averaging methods. In vapor phase samples, dry deposition samples, dissolved and particulate tributary water samples, there was almost no difference between median and mean plots (Figures 1.6.11 - 1.6.14). In wet deposition samples, dissolved and particulate water samples, surficial sediment samples, and 5-6 year-old lake trout samples, there were some small differences in the plots (Figures 1.6.15 - 1.6.19), with median values being somewhat lower in all instances due to the fact that some of the congeners have zero median values. These zero median values were the result of a large number of congeners with reported values of zero or near zero. We decided to use mean values for the rest of the analysis because there appeared to be little difference between median and mean plots and to avoid the zero median values.

1.6.4.3 Comparison of Congener Patterns in Different Media in Segment 2/Saugatuck Biota Box

When the congener patterns for different media in segment 2 and the Saugatuck biota box were compared (Figure 1.6.20), the following general trends were identified. Vapor phase data appeared to have a higher percentage of lower chlorinated congeners than any of the other media. Dissolved PCB congeners in water from the Kalamazoo River most nearly matched the congener pattern seen in the vapor phase, with the congener pattern for dichloro, tetrachloro — decachloro's most closely

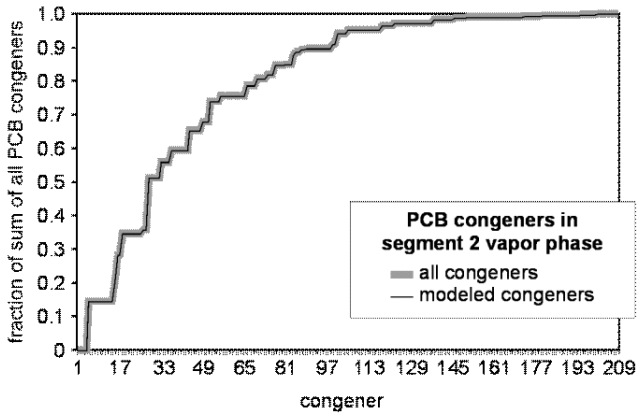

Figure 1.6.2. Cumulative frequency distribution – PCB congeners in segment 2 vapor phase.

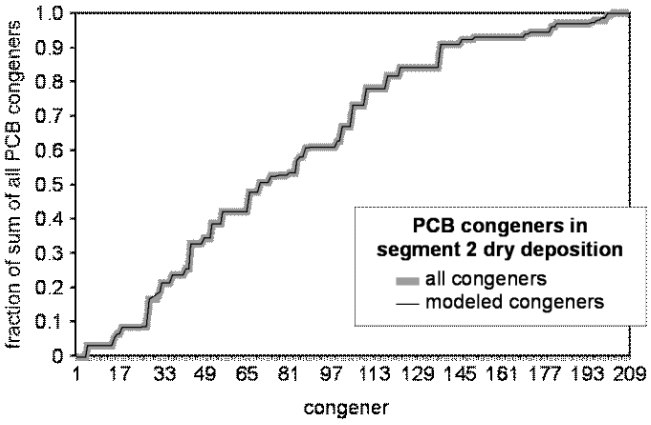

Figure 1.6.3. Cumulative frequency distribution – PCB congeners in segment 2 dry deposition.

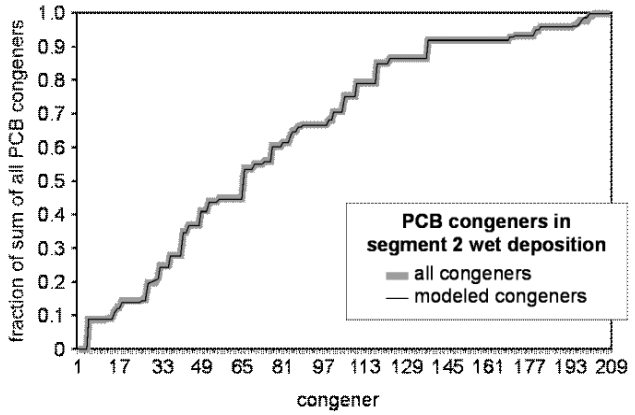

Figure 1.6.4. Cumulative frequency distribution – PCB congeners in segment 2 wet deposition.

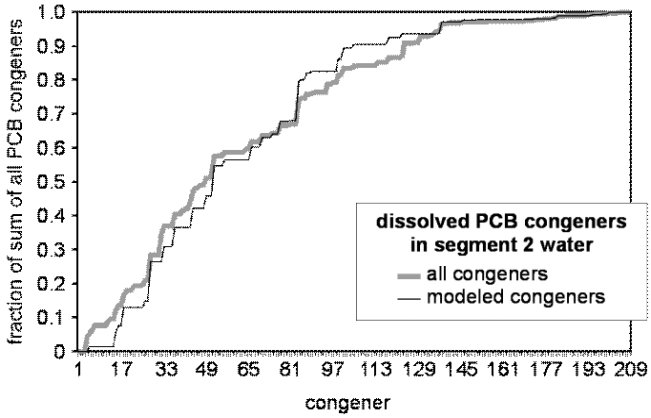

Figure 1.6.5. Cumulative frequency distribution – dissolved PCB congeners in segment 2 water.

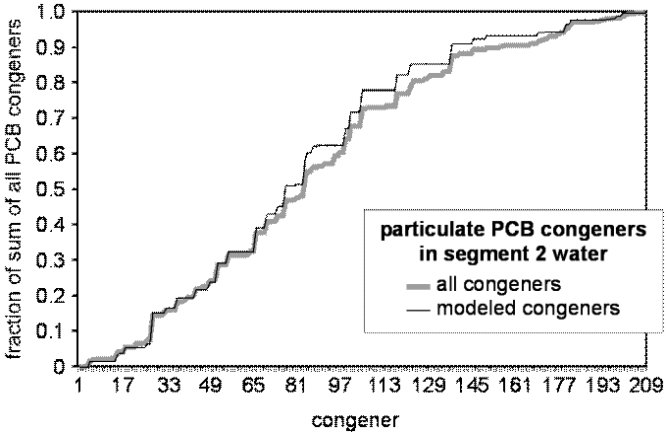

Figure 1.6.6. Cumulative frequency distribution – particulate PCB congeners in segment 2 water.

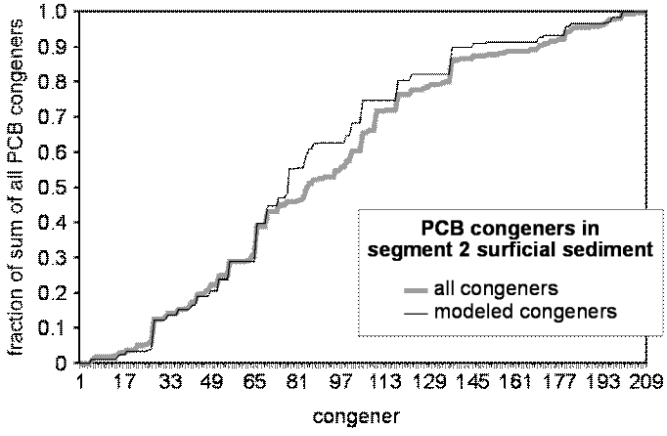

Figure 1.6.7. Cumulative frequency distribution – PCB congeners in segment 2 surficial sediment.

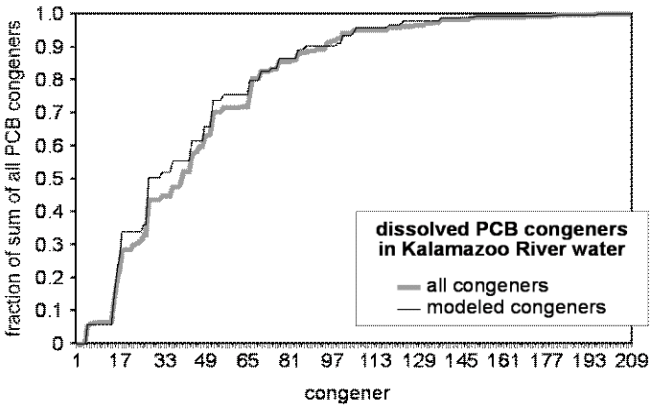

Figure 1.6.8. Cumulative frequency distribution – dissolved PCB congeners in Kalamazoo River water.

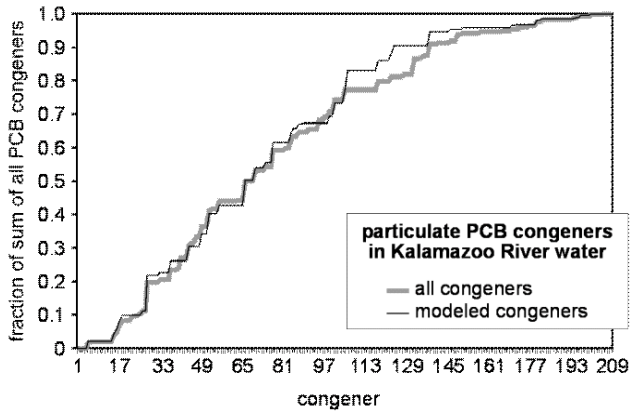

Figure 1.6.9. Cumulative frequency distribution – particulate PCB congeners in Kalamazoo River water.

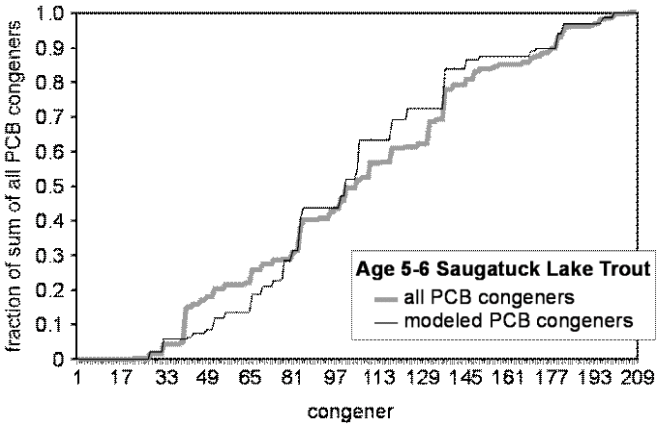

Figure 1.6.10. Cumulative frequency distribution – age 5-6 Saugatuck lake trout.

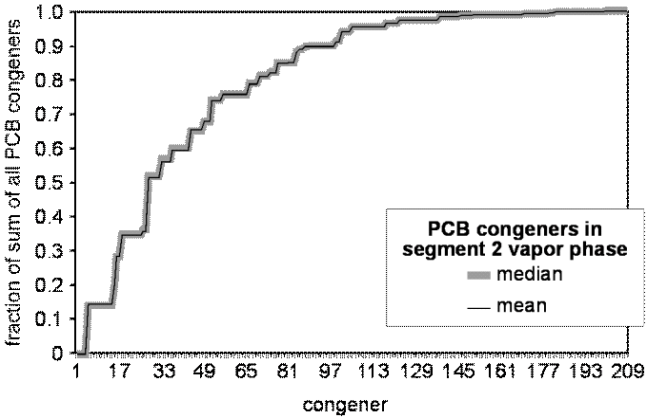

Figure 1.6.11. Cumulative frequency distribution – PCB congeners in segment 2 vapor phase.

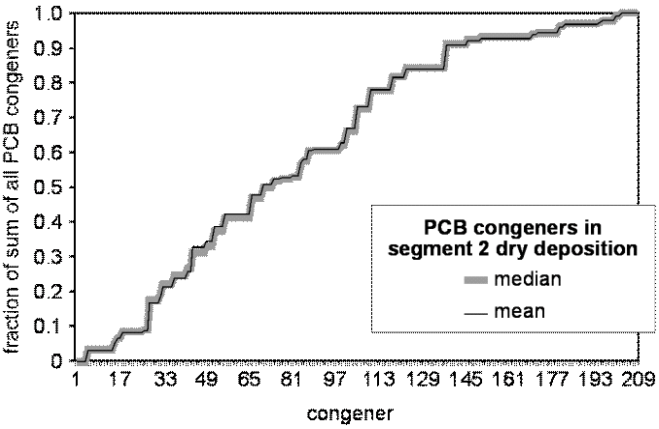

Figure 1.6.12. Cumulative frequency distribution – PCB congeners in segment 2 dry deposition.

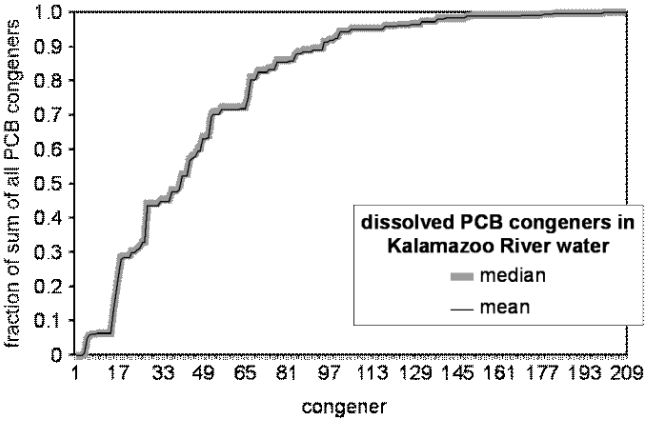

Figure 1.6.13. Cumulative frequency distribution – dissolved PCB congeners in Kalamazoo River water.

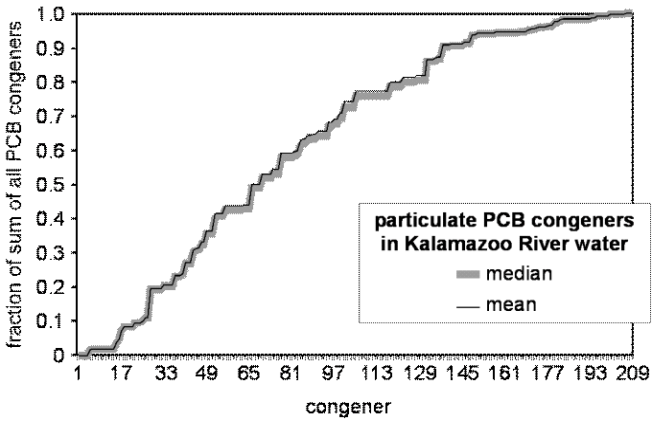

Figure 1.6.14. Cumulative frequency distribution – particulate PCB congeners in Kalamazoo River water.

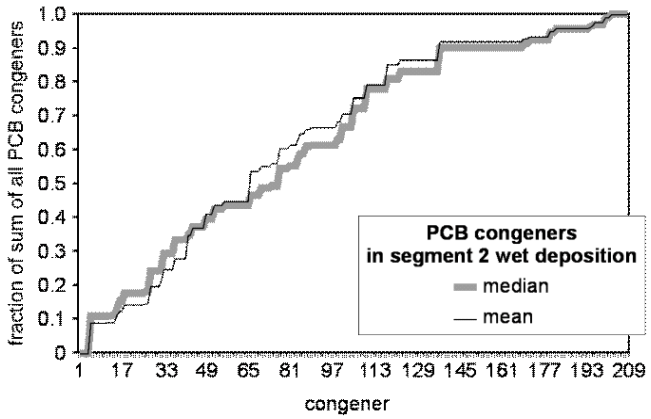

Figure 1.6.15. Cumulative frequency distribution – PCB congeners in segment 2 wet deposition.

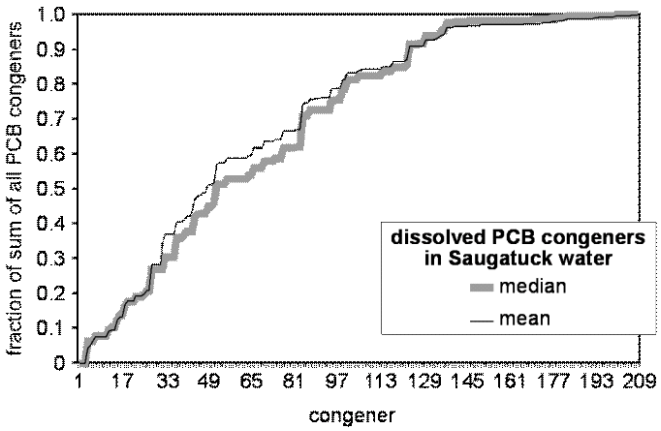

Figure 1.6.16. Cumulative frequency distribution – dissolved PCB congeners in Saugatuck water.

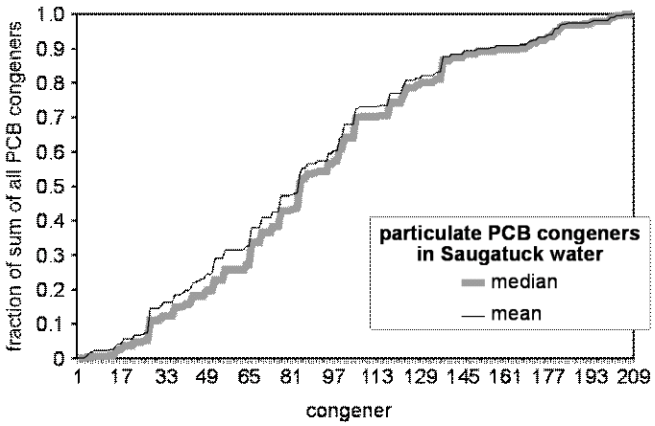

Figure 1.6.17. Cumulative frequency distribution – particulate PCB congeners in Saugatuck water.

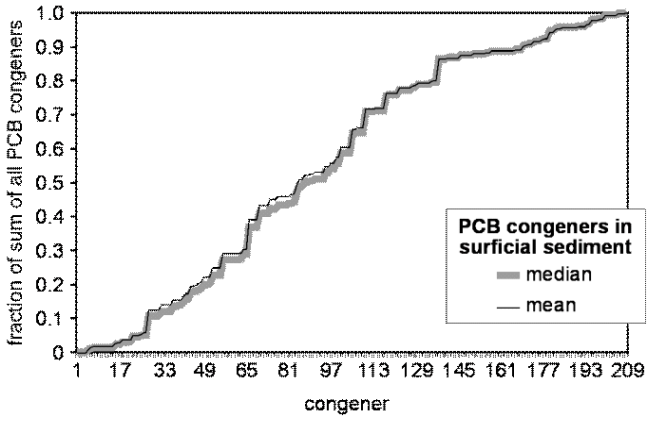

Figure 1.6.18. Cumulative frequency distribution – PCB congeners in surficial sediment.

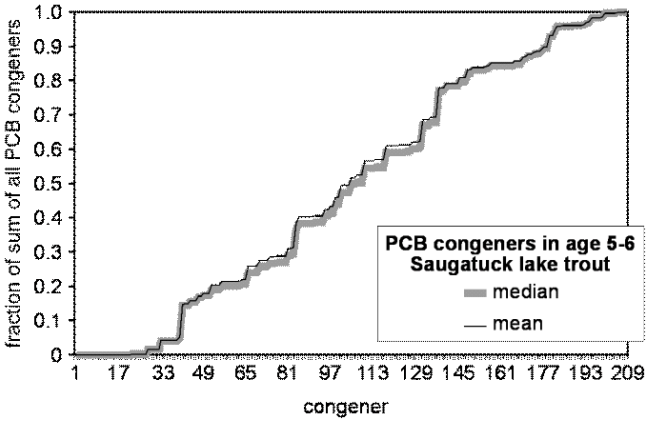

Figure 1.6.19. Cumulative frequency distribution – PCB congeners in age 5-6 Saugatuck lake trout.

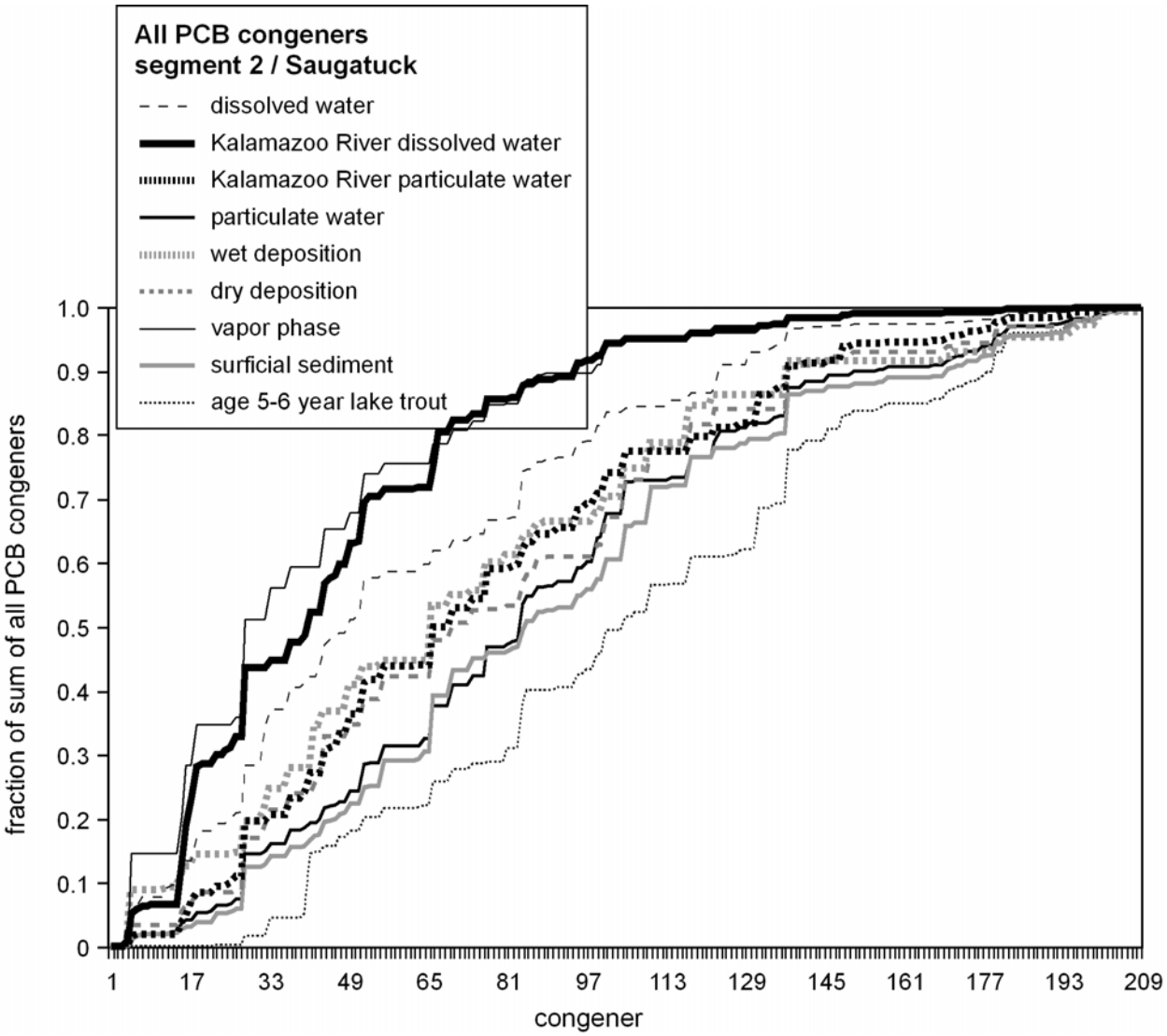

Figure 1.6.20. PCB congeners in segment 2, Saugatuck.

matching the vapor phase data. The pattern seen for open lake dissolved water samples did not closely match any of the other media. Open lake dissolved PCB congeners in water had a lower percentage of lower chlorinated congeners than either the vapor phase or dissolved water congener samples from the Kalamazoo River, but it had a higher percentage of these congeners than any of the other media.

The Kalamazoo River particulate PCB congeners in water had a higher percentage of lower congeners than open lake particulate PCB congeners in water and closely matched the congener patterns seen in both wet and dry deposition data for segment 2. Open lake particulate PCB congeners data most closely matched the surficial sediment for segment 2. Age 5-6 year-old lake trout had a much lower percentage of lower chlorinated congeners than any of the other media.

1.6.4.4 Comparison of Atmospheric Congener Data

Atmospheric PCB samples for vapor phase and wet and dry deposition media were collected from a number of land-based stations around Lake Michigan, as well as from shipboard stations (Figure 1.6.21) on several LMMBP surveys (U.S.

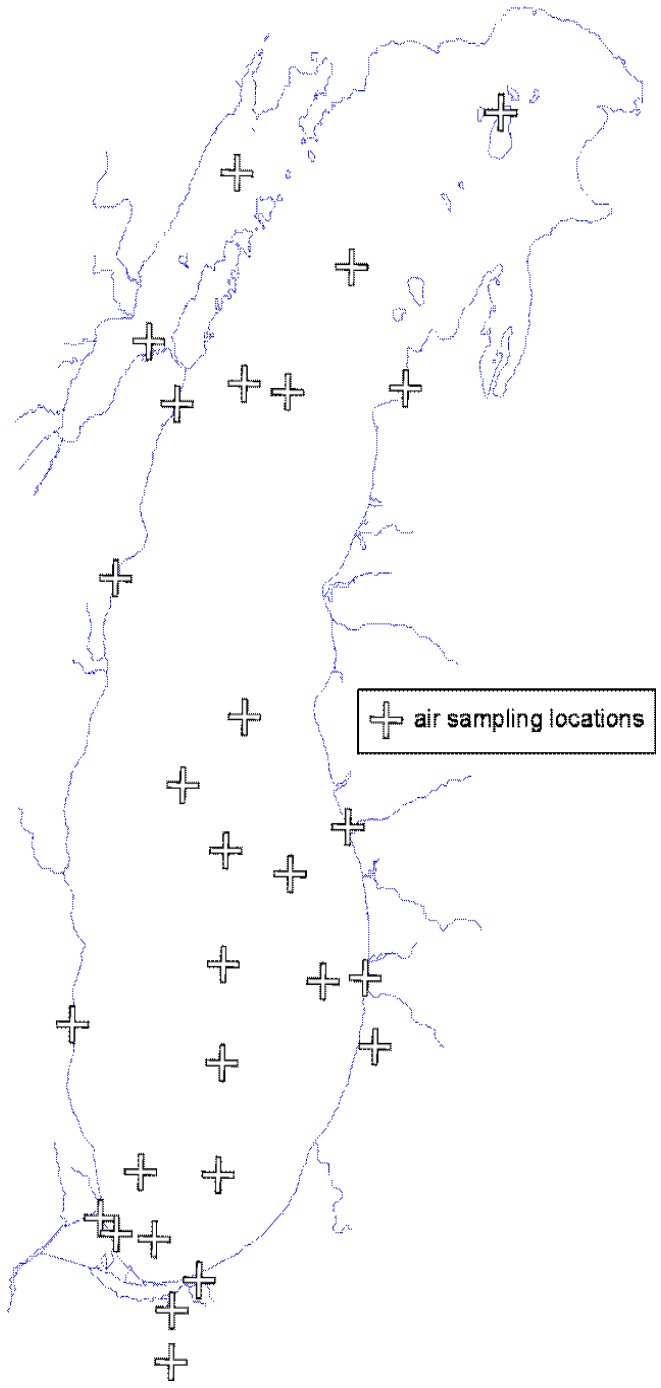

Figure 1.6.21. Air sampling locations.

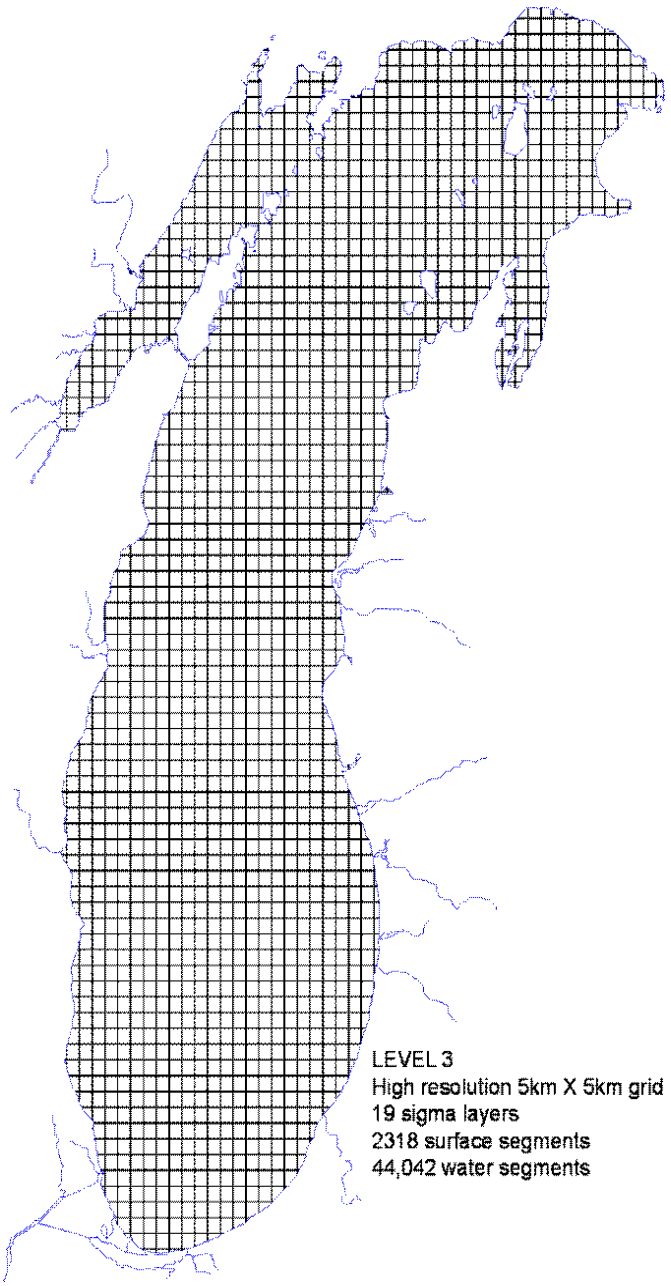

Figure 1.6.22. Lake Michigan high-resolution 5 km x 5 km grid with 19 sigma layers.

Environmental Protection Agency, 1997). These data were then used by Hornbuckle to generate over-water concentrations of PCB congeners for all 5 km surface grid cells used for the high-resolution model (Green, 2000; Miller *et al.*, 2001) (see Figure 1.6.22). Hornbuckle only analyzed modeled congeners, thus the congener set for this media is somewhat smaller than in other media. These data were then aggregated for modeling into surface segment cells for the LM2-Toxic model (Figure 1.6.1). As stated previously, the Saugatuck biota box (LMMBP-Biota3) is located within LM2 segment 2. A congener pattern comparison was done to see if the congener patterns

seen in segment 2 were similar to patterns seen in other segments. This was indeed the case with vapor phase and wet deposition data (Figures 1.6.23 and 1.6.24). For dry deposition samples, LM2 segment 1, which is the southwest corner of the lake, is somewhat lower in tetrachloro and pentachloro congeners than all of the other segments, which have a very similar congener pattern (Figure 1.6.25).

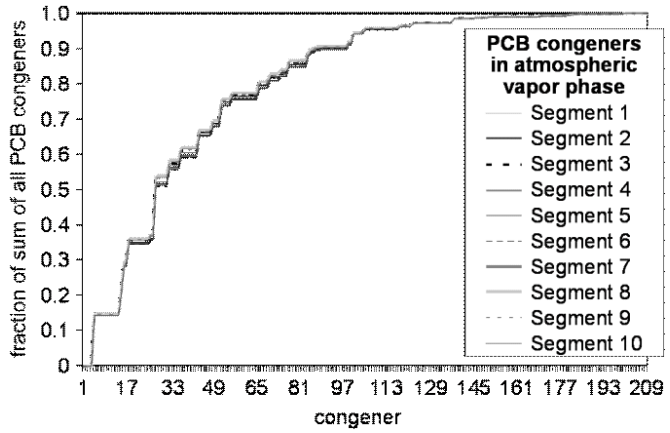

Figure 1.6.23. Cumulative frequency distribution (mean) – PCB congeners in atmospheric vapor phase.

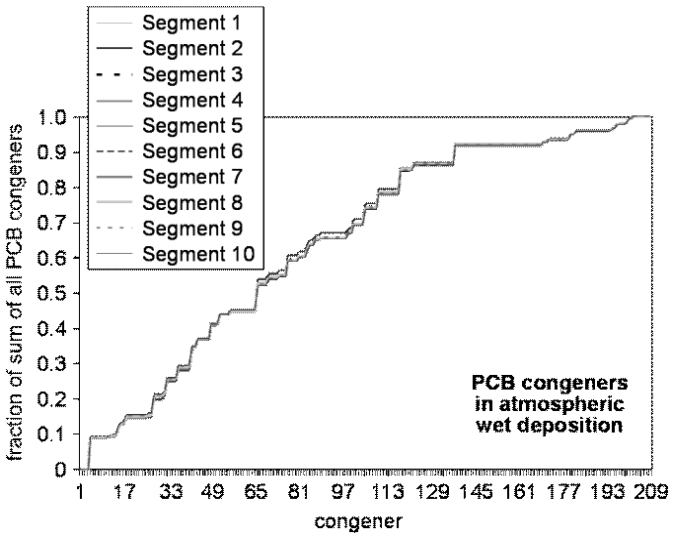

Figure 1.6.24. Cumulative frequency distribution (mean) – PCB congeners in atmospheric wet deposition.

A comparison of vapor phase PCB samples from segment 2 to wet and dry deposition samples from this segment (Figure 1.6.26) showed a somewhat similar congener distribution for wet and dry

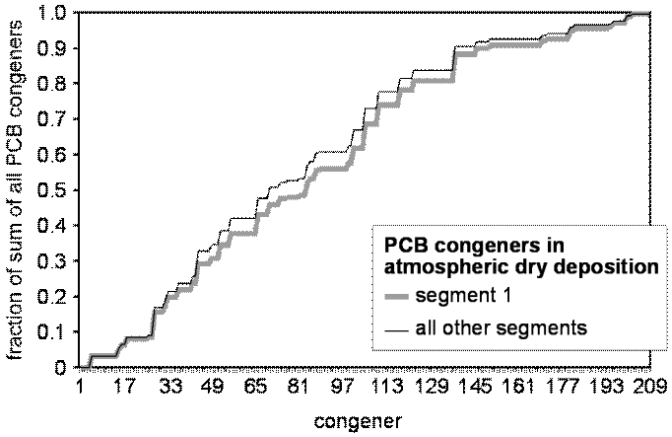

Figure 1.6.25. Cumulative frequency distribution (mean) – PCB congeners in atmospheric dry deposition.

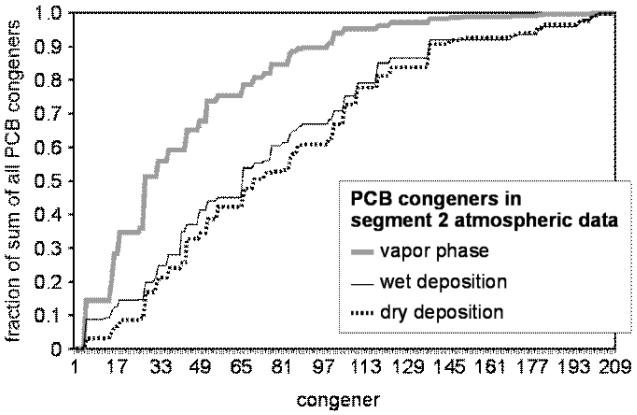

Figure 1.6.26. Cumulative frequency distribution (mean) – PCB congeners in segment 2 atmospheric data.

deposition samples. The vapor phase congener pattern was quite different with a much higher percentage of lower chlorinated congeners.

1.6.4.5 Comparison of Tributary Congener Patterns

An examination of 11 monitored LMMBP tributaries show that dissolved water samples clearly had a higher percentage of lower chlorinated congeners than do particulate water samples (Figure 1.6.27). The Fox River dissolved PCBs in water had a higher percentage of lower congeners than any of the other dissolved tributary samples. This same trend held true for Fox River particulate water samples.

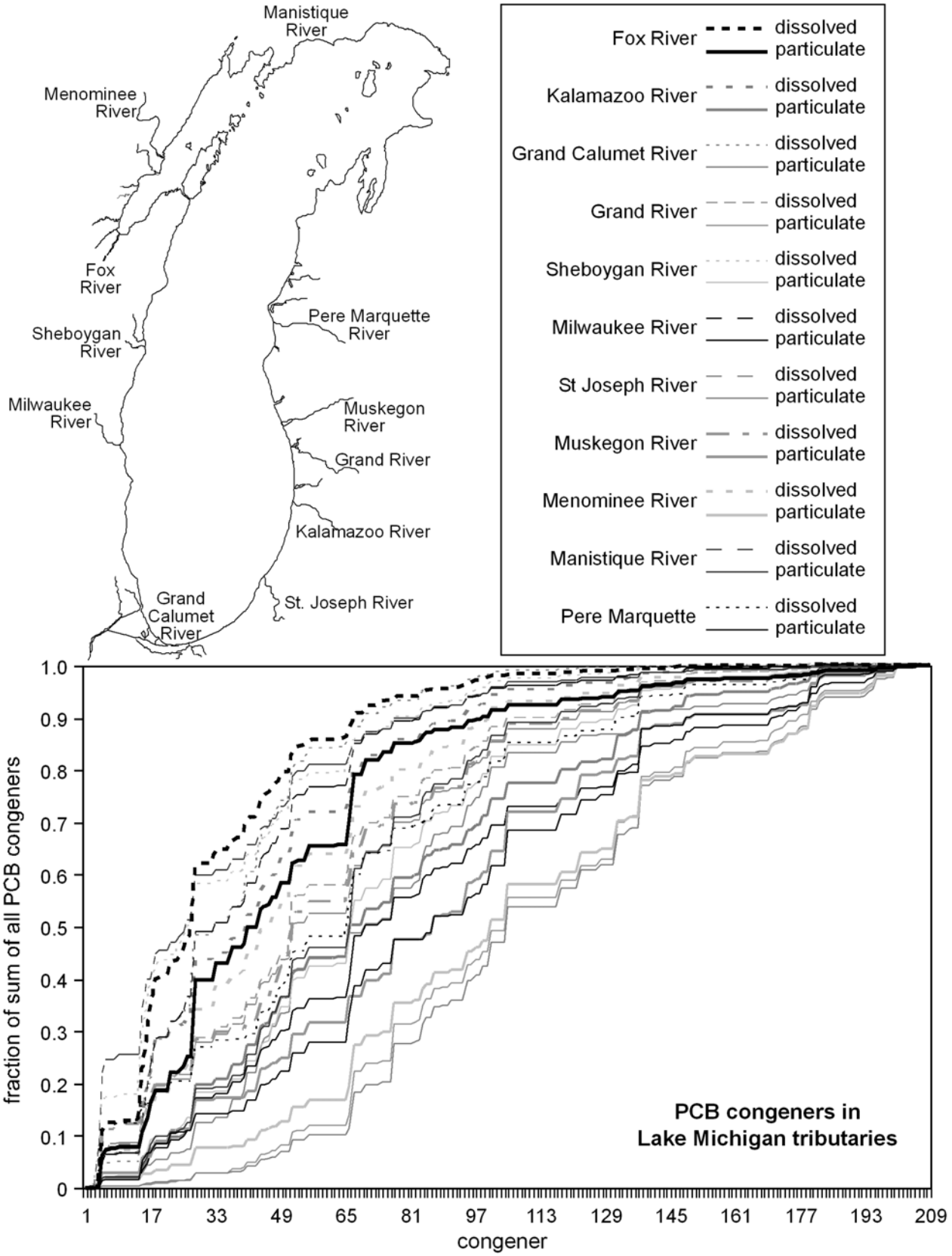

Figure 1.6.27. PCB congeners in Lake Michigan tributaries.

When segment 2 data for other media were overlaid on the tributary data, it appears that vapor phase data most closely matched dissolved water tributaries data from the western side of the lake (Figure 1.6.28), dissolved water data from segment 2 most closely matched dissolved water tributary data from rivers in the lower peninsula of Michigan (Figure 1.6.29), and particulate water data from segment 2 generally fell in the middle of the particulate tributary data (Figure 1.6.30).

1.6.4.6 Comparison of Ages 5 and 6 Lake Trout Congener Patterns in All Biota Boxes

Five and six year-old lake trout were collected and analyzed from all three LMMBP biota boxes (Sturgeon Bay: LMMBP-Biota1, Sheboygan Reef: LMMBP-Biota2, and Saugatuck: LMMBP-Biota3). Congener patterns were similar for all three biota boxes, with Saugatuck having a somewhat higher percentage of lower congeners, Sheboygan reef having the lowest percentage of lower congeners, and Sturgeon Bay being in between (Figure 1.6.31).

1.6.4.7 Comparison of Different Lake Trout Age Class Congener Patterns in Saugatuck Biota Box

A comparison was done between different age classes of lake trout in the Saugatuck biota box to see if the congener patterns were different for different age classes. Lake trout data for year classes 2, 3, and 9 were compared to age classes 5 and 6 lake trout (Figure 1.6.32). This comparison showed that there was very little difference between the congener patterns of these different age classes.

1.6.5 Conclusions

When all media were looked at, there were very clear differences in the congener patterns that were observed.

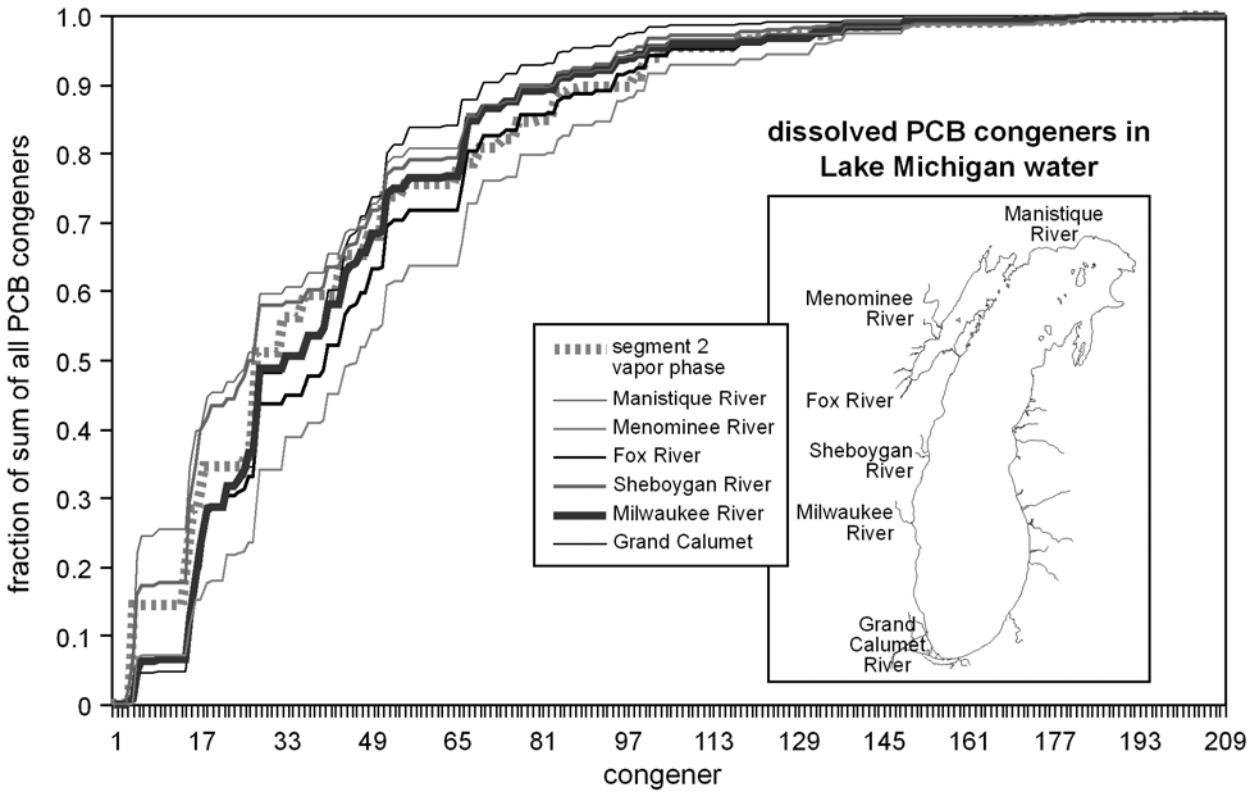

Figure 1.6.28. Comparison of dissolved PCB congeners in Lake Michigan western tributaries to segment 2 vapor phase.

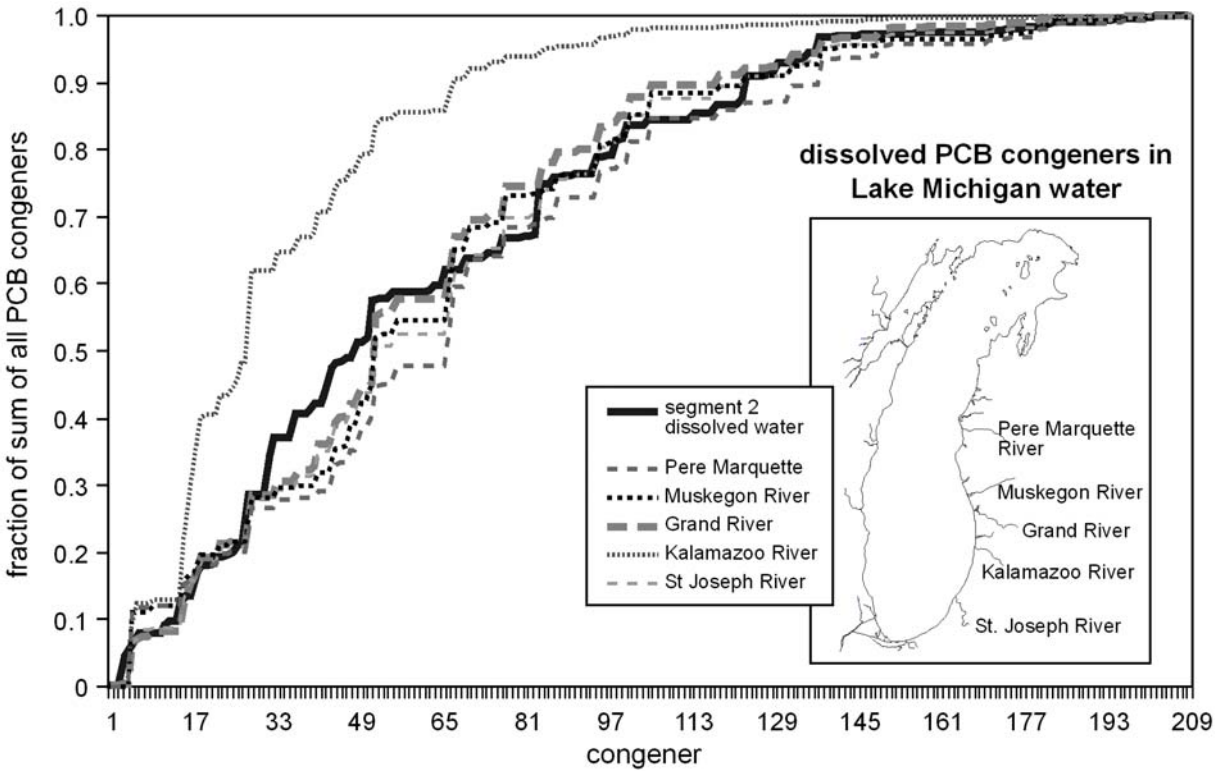

Figure 1.6.29. Comparison of dissolved PCB congeners in Lake Michigan eastern tributaries to segment 2 water.

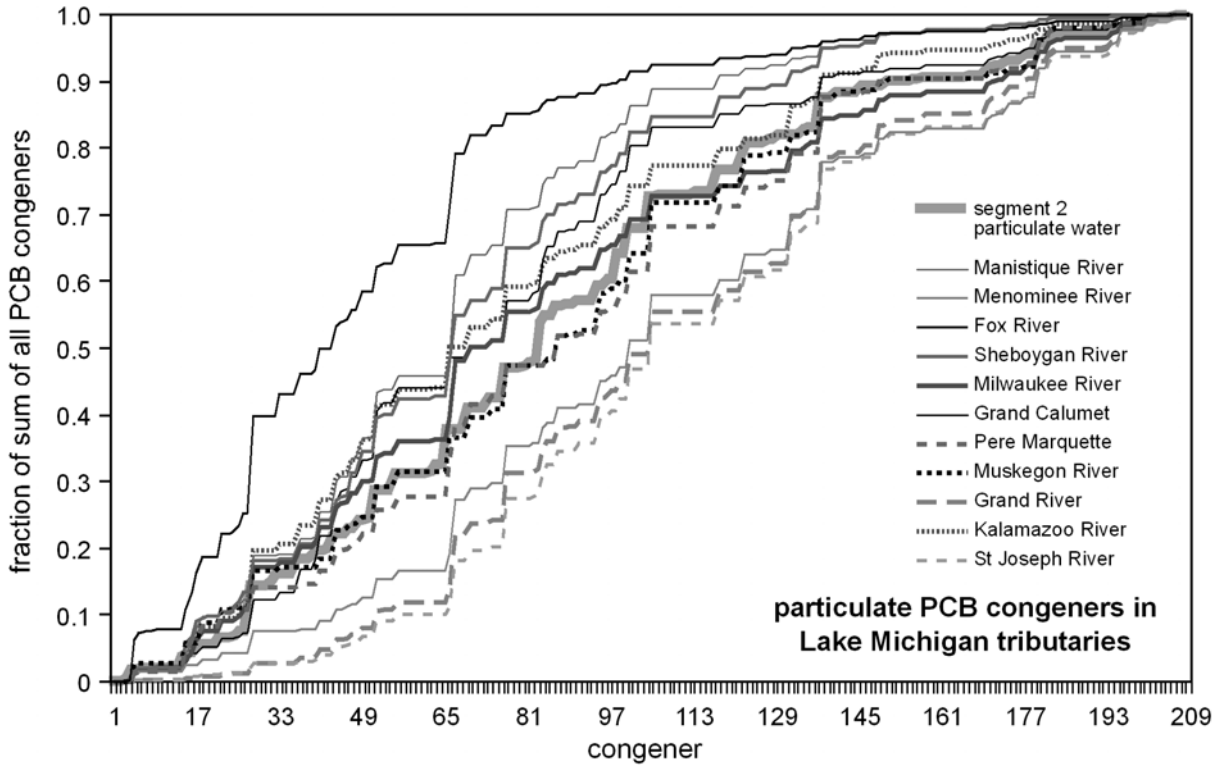

Figure 1.6.30. Comparison of particulate PCB congeners in Lake Michigan segment 2 to tributaries.

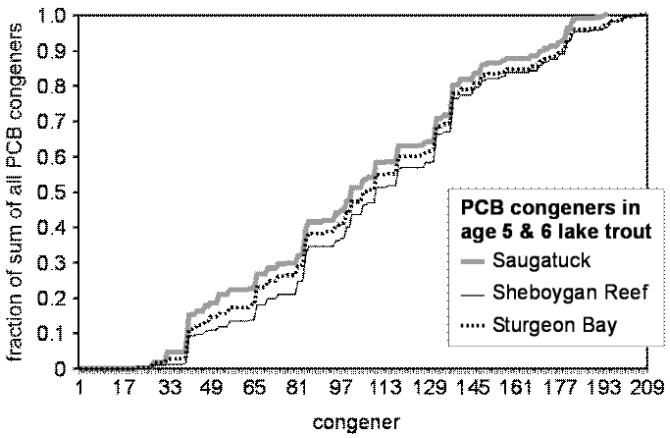

Figure 1.6.31. Cumulative frequency distribution (mean) – PCB congeners in ages 5 and 6 lake trout.

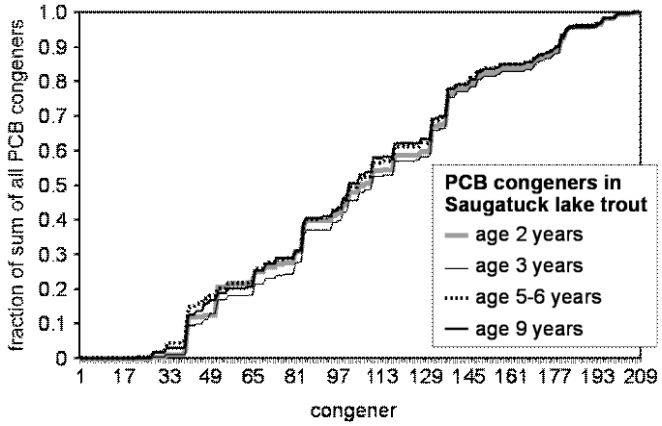

Figure 1.6.32. Cumulative frequency distribution (mean) – PCB congeners in Saugatuck lake trout.

Vapor phase data had the highest percentage of lower chlorinated congeners of all the media examined. In all cases, the dissolved fraction of water samples had a higher percentage of lower chlorinated congeners than the corresponding particulate fraction of water samples. The Kalamazoo River dissolved and particulate water samples had a higher percentage of lower chlorinated congeners than the corresponding open lake dissolved and particulate water samples. The Kalamazoo River dissolved water samples most closely resembled the vapor phase data, while the Kalamazoo River particulate water data most closely resembled segment 2 wet and dry deposition data. This could be due to the possibility that PCBs in the river are from a newer (non-weathered) source of contamination than open lake samples.

Open lake particulate samples most closely matched the surficial sediment data for segment 2. This result probably is not too surprising considering the close relationship between sediments and particulates and the continuous deposition and resuspension of bottom sediments in the water column.

Atmospheric congener patterns were very uniform over the entire lake, with the exception of dry deposition data which had a somewhat different congener patten in segment 1 than in all of the other model segments. Wet and dry deposition samples showed similar congener patterns for segment 2 with dry deposition samples having a slightly lower congener distribution pattern than wet deposition samples. This is the same trend that was seen in lake water and tributary samples. Vapor phase samples for segment 2 had a very different congener pattern with a much higher percentage of lower chlorinated congeners.

Dissolved tributary water samples had a higher percentage of lower chlorinated congeners than particulate water samples. It is interesting that dissolved water tributary data, with the exception of the Kalamazoo River, from the western side of Lake Michigan (Fox, Grand Calumet, Sheboygan, Milwaukee, Manistique, and Menominee Rivers) generally had a higher concentration of the lower chlorinated congeners than tributaries from the lower peninsula of Michigan (Grand, Muskegon, Pere Marquette, and St. Joseph Rivers) (Figure 1.6.33). This same trend generally holds true for the tributary particulate data (Figure 1.6.34). Overall, the Fox River had the highest percentage of lower chlorinated congeners for both dissolved and particulate samples.

Fish from all three biota boxes had very similar congener patterns. In addition, fish from different age classes in the same biota box (Saugatuck) also had very similar congener patterns. This would seem to indicate that the same processes are responsible for determining the congener pattern distribution in fish. This pattern was also similar to the trend that Kuehl observed in her analysis of LMMBP biota samples (Kuehl, 2002), where she reported that

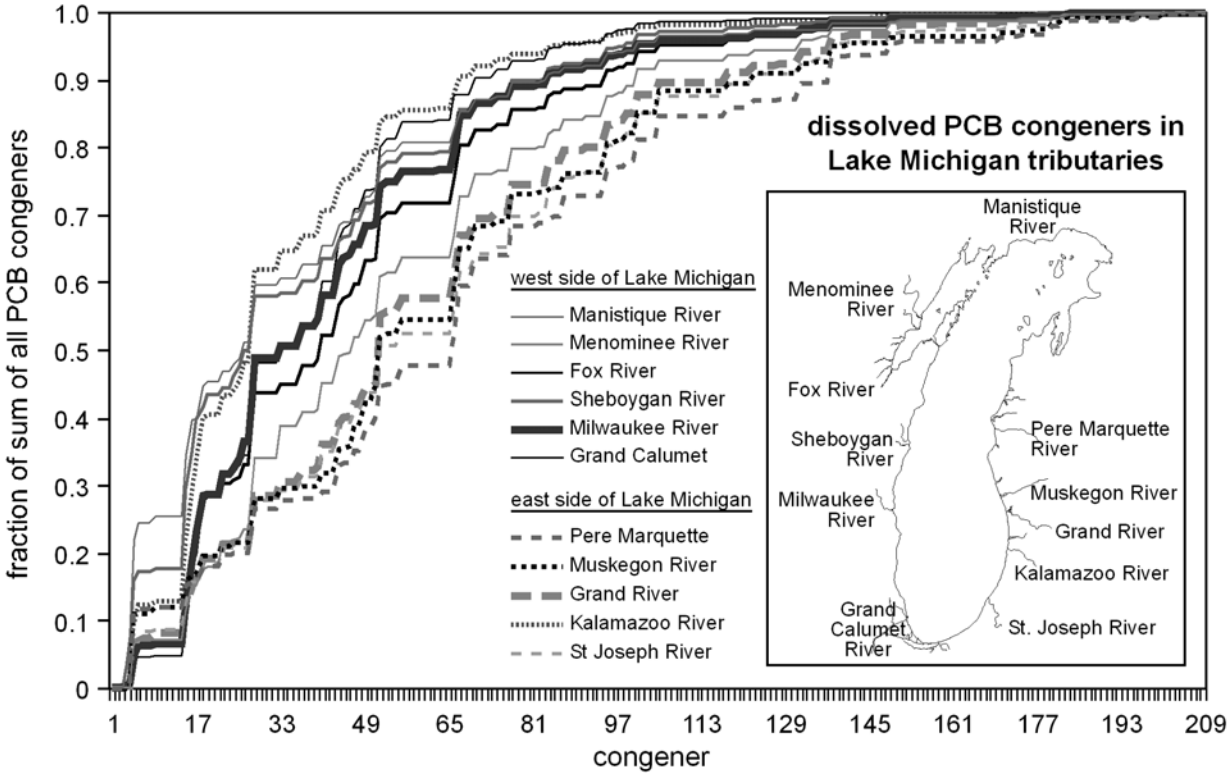

Figure 1.6.33. Comparison of dissolved PCB congeners in west side-to-east side of Lake Michigan tributaries.

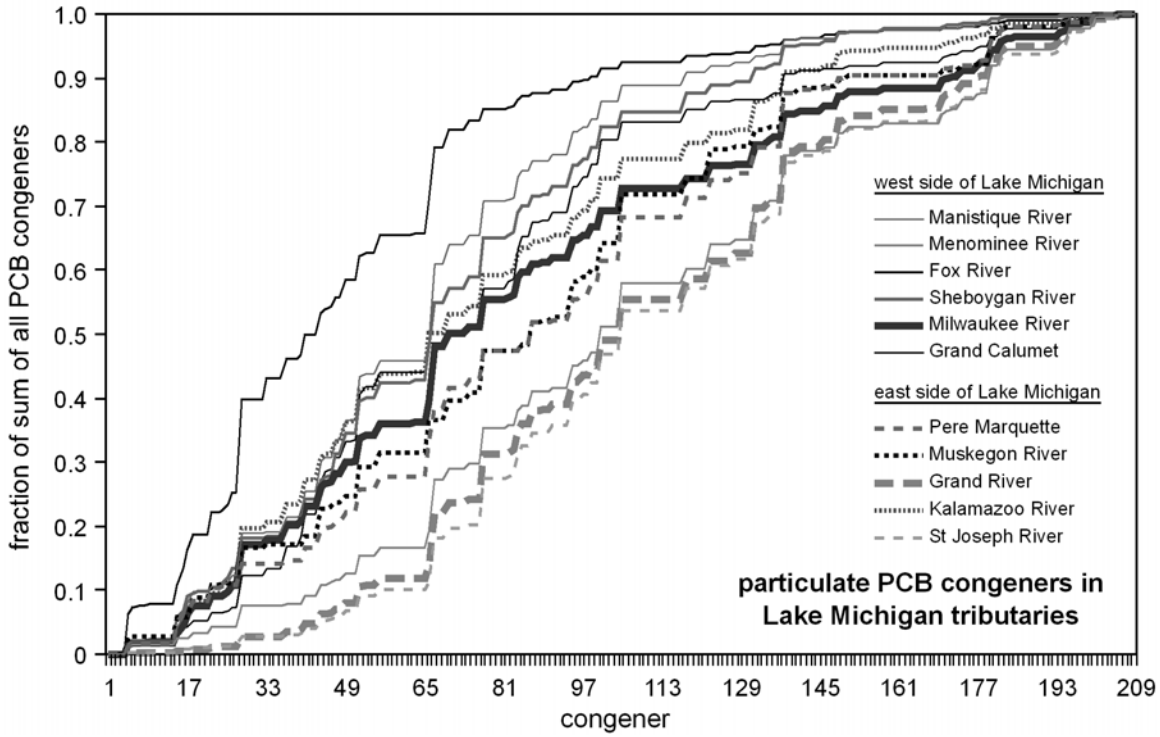

Figure 1.6.34. Comparison of particulate PCB congeners in west side-to-east side of Lake Michigan tributaries.

"With the exception of deepwater sculpin samples collected in Biota Box #3 near Saugatuck, all LMMBP biota sample congener patterns were homogeneous", and "A relatively consistent pattern of the selected PCB congeners was measured in all of the biota species or classifications." Also, while the age class 5-6 year-old lake trout congeners pattern for biota box number 3 most nearly matched the congener patterns of surficial sediment and particulate water samples from modeling segment 2, this was not a very close match.

From this data investigation, it was clear that it was impossible to relate PCB contamination of lake trout to a tributary source based on congener pattern matching. This is due to the fact that tributary congener patterns for Lake Michigan, regardless of source, had congener patterns which were significantly different from all lake trout congener patterns. In addition, congener patterns seen in lake trout from different biota boxes had very similar congener patterns, which would indicate that there are no spatial differences in lake trout congener patterns.

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